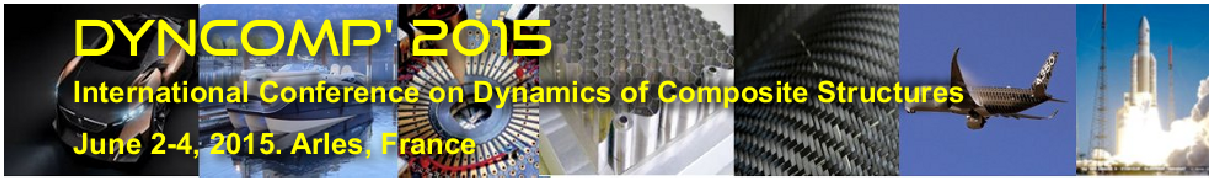

DYNAMIC MECHANICAL ANALYSIS OF CARBON-CERAMIC COMPOSITES

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ABSTRACT

The aim of this work is to compare mechanical behavior of phenol-formaldehyde resin-derived CC (carbon) composite with CC/ceramic (carbon/ceramic) composites obtained by the impregnation of CC composite with commercially available polysiloxane-based solutions of preceram and their subsequent heat treatment at 1000 °C, 1500 °C and 1700 °C. CC/ceramic composites heat treated at 1000 °C and 1500 °C contain silicon oxycarbide [2] and CC/ceramic composite heat treated at 1700 °C contains silicon carbide [2]. As a reinforcement HTS 5131 carbon fibers (Tenax) in a form of roving were used. Phenol-formaldehyde resin (Organika-Sarzyna, Poland) and Lukosil 901 polysiloxane substrate (Lucebni zavody, Czech Republic) used in this experiments were inexpensive (cost about 10$/kg).

Dynamic mechanical analysis (DMA) in three-point clamping mode was carried out. All tests were carried out in air at 450 °C at an oscillatory frequency of 20 Hz. A multifrequency-strain mode was set up under a strain of 80 μm. CC composite was used as a reference.

In comparison to the reference CC composite, CC/ceramic composites exhibited up to 10 times longer lifetime.

1 INTRODUCTION

Carbon fibres-reinforced carbon composites (CC composites) are candidate materials for advanced structures, which could work under dynamic load at elevated temperature. Their mechanical properties are retained even until 2000 °C, and due to low values of the coefficients of thermal expansion (CTE) and high heat of sublimation they have good ablation resistance.

Additionally, CC composites exhibit thermal shock resistance and chemical resistance in non-oxidizing atmosphere.

However, application of CC composites in high temperature structures is limited due to the potential oxidation damage and erosion in air above 400 ^{o}C [1]. Much effort is done in order to protect CC composites against air – oxidation. Several methods have been developed to improve oxidation resistance: chemical vapor deposition (CVD) coatings, multilayer coatings, impregnation of CC composite with organosilicon compounds, i.e. polysiloxanes and heat treatment [1]. A cross-linked polysiloxane resin during pyrolysis up to 1000 ^{o}C can be transformed into a silicon oxycarbide (structure containing Si-C-O bonds, blackglass), and during pyrolysis up to 1700 ^{o}C silicon carbide crystallizes [2]. Our previous work indicate that depending on the structure of polysiloxane resins it is possible to obtain ceramic samples with high ceramic yield, i.e. from 82 to 86 wt % at 1000 ^{o}C and from 61 to 70 wt % at 1700 ^{o}C [2].

The aim of this work is to compare dynamic mechanical properties of phenol-formaldehyde resin-derived CC (carbon) composite with CC/ceramic (carbon/ceramic) composites obtained by the impregnation of CC composite with commercially available polysiloxane-based solutions of preceram and their subsequent heat treatment up to 1700 ^{o}C. As a reinforcement carbon fibers in a form of roving were used. Phenol-formaldehyde resin and polysiloxane substrate used in this experiments were inexpensive (cost about 10$/kg).

2 MATERIALS AND METHODS

As a reinforcement HTS 5131 carbon fibers (Tenax) in a form of roving were used. To prepare the unidirectional fiber prepreg tapes the carbon fibers were impregnated with phenol-formaldehyde resin (Organika-Sarzyna, Poland). The tapes were dried and cut to obtain 15 cm long laminates and unidirectionally stacking laminates were placed in a metallic mold. The stacked layup was heated up to 140 ^{o}C in air atmosphere under a pressure of 10 MPa. Then, the composite samples were heated to 1000 ^{o}C in an argon atmosphere to obtain CC composite. C/C composites obtained in such a way were impregnated with Lukosil 901 polysiloxane (PS) resin (Lucebni zavody, Czech Republic). Impregnated composite samples were subjected to subsequent thermal treatment in an inert argon atmosphere. CC/ceramic composites heat treated at 1000 ^{o}C and 1500 ^{o}C contain silicon oxycarbide [2] and CC/ceramic composite heat treated at 1700 ^{o}C contains silicon carbide [2]. Samples in a form of bars were prepared (1mm x 4mm x 35mm).
Types of prepared CC/ceramic composites are presented in Table 1, the characteristics of the composites are presented in Table 2 and their mechanical properties are presented in Table 3.

	Composite	Description	Matrix
Reference	CC composite	CC	C
Sample 1	CC/ceramic composites	CC + PS, HT 1000 ^{o}C	C+$SiCO_a$
Sample 2		CC + PS, HT 1500 ^{o}C	C+$SiCO_b$
Sample 3		CC + PS, HT 1700 ^{o}C	C+SiC

Table 1. Types of prepared composites

Composite	Aparent density [g/cm^3]	Specific density [g/cm^3]	Porosity [%]
Reference	1.18	1.53	23
Sample 1	1.39	1.75	14
Sample 2	1.33	1.63	20
Sample 3	1.19	1.56	21

Table 2. Microstructure of composites

Composite	Bending strength [MPa]	Young's modulus [GPa]	Deflection [mm]	ILSS [MPa]
Reference	300 ± 20	91 ± 6	0.73 ± 0.15	14 ± 2
Sample 1	287 ± 11	71 ± 7	0.85 ± 0.15	15 ± 1
Sample 2	289 ± 15	74 ± 14	0.82 ± 0.02	14 ± 1
Sample 3	279 ± 32	78 ± 14	0.55 ± 0.02	15 ± 1

Table 3. Mechanical properties of composites

Dynamic mechanical analysis (DMA) in three-point clamping mode was carried out with TA Q800 DMA analyzer equipped with a TA Universal Analysis 2000 software. The span between supports was 20 mm. All tests were carried out in air at 450 °C at an oscillatory frequency of 20 Hz. A multifrequency-strain mode was set up under a strain of 80 µm. CC composite was used as a reference.

3 RESULTS AND DISCUSSION

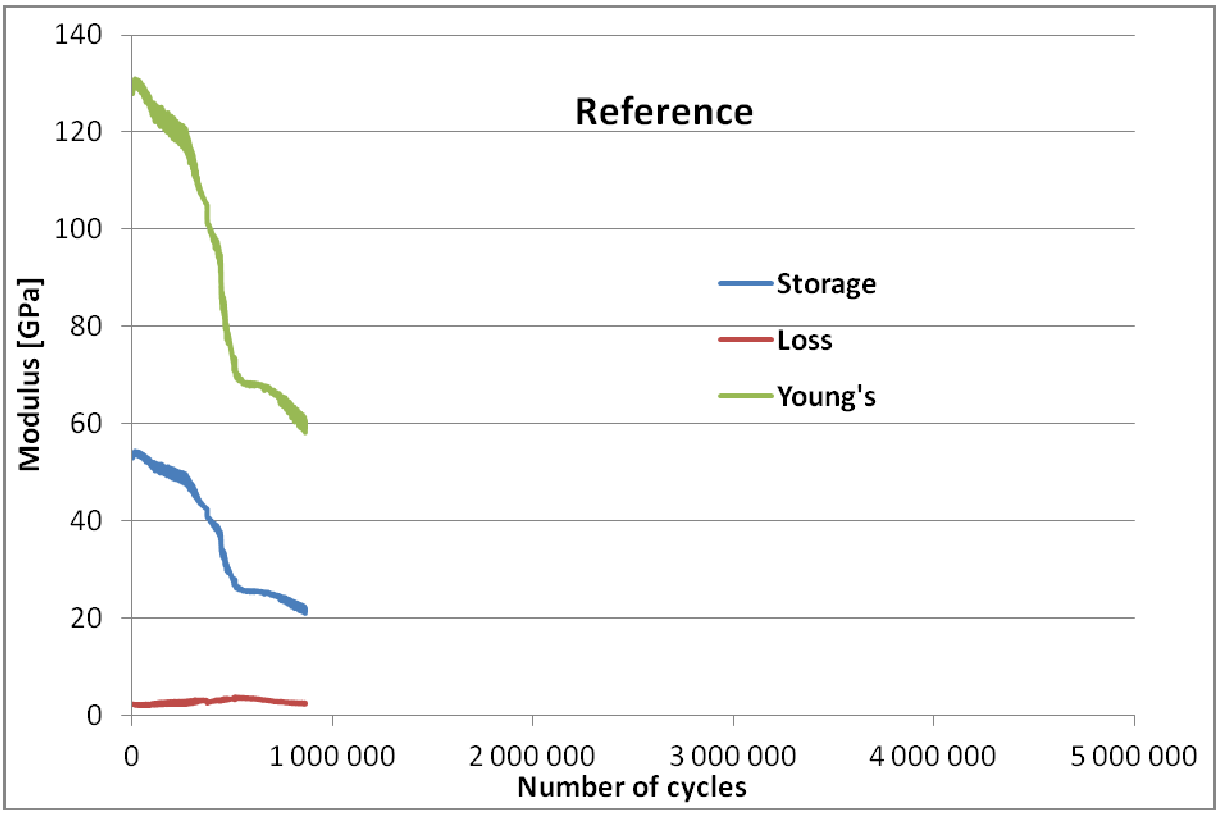

Fig. 1. Results of DMA for reference sample

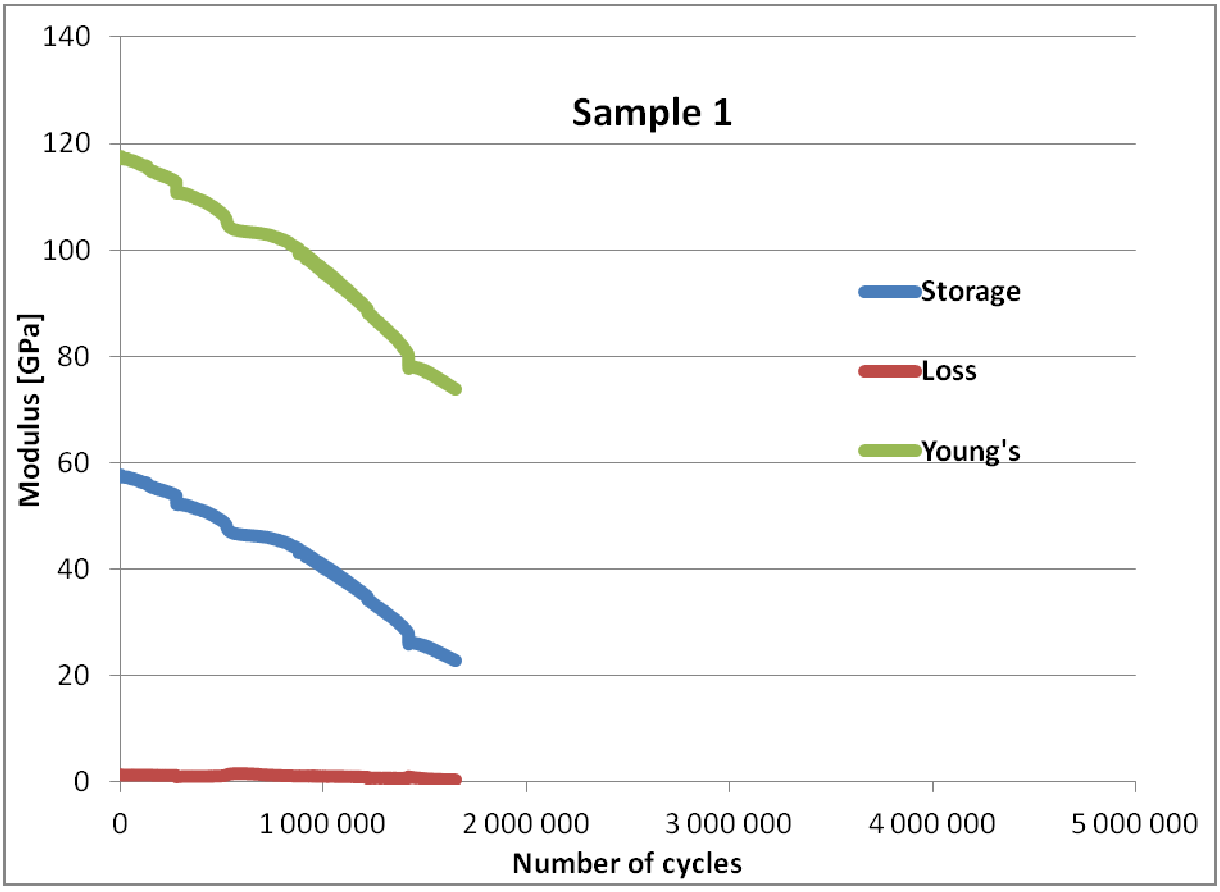

Fig. 2. Results of DMA for sample 1

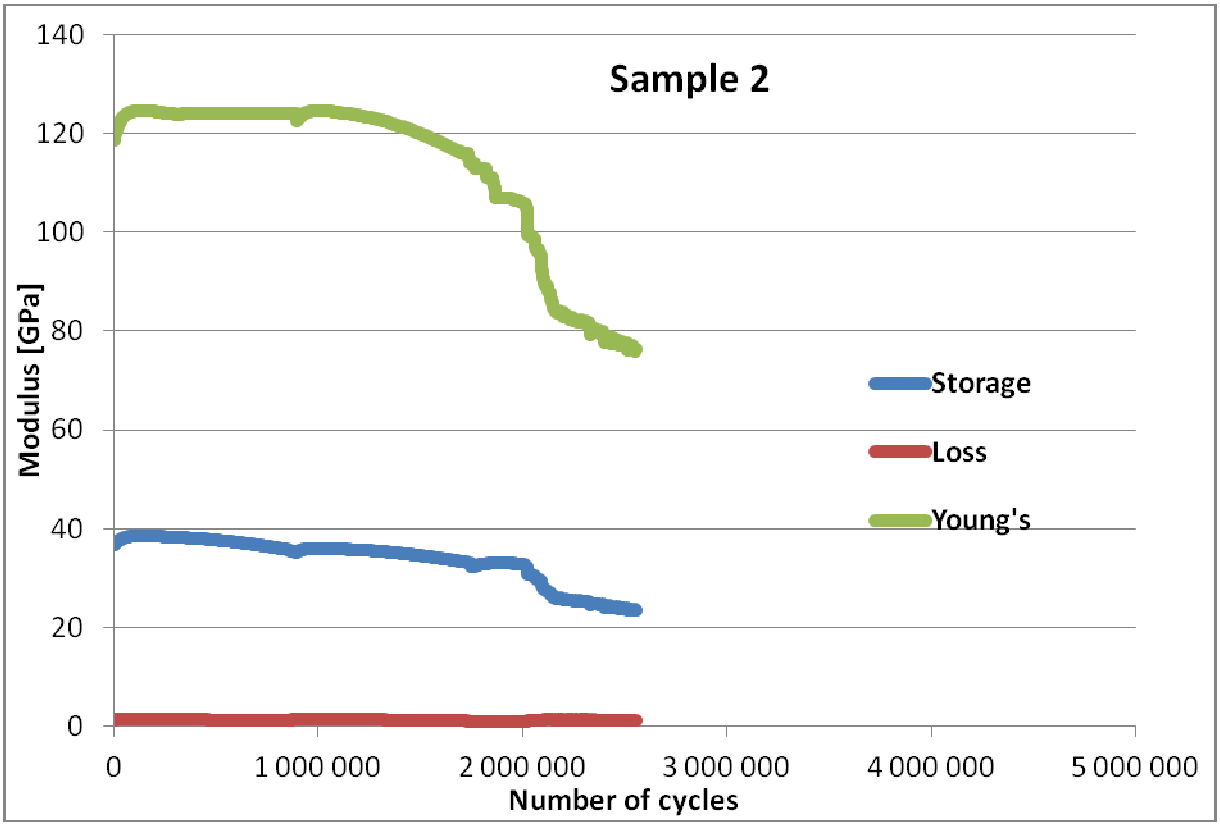

Fig. 3. Results of DMA for sample 2

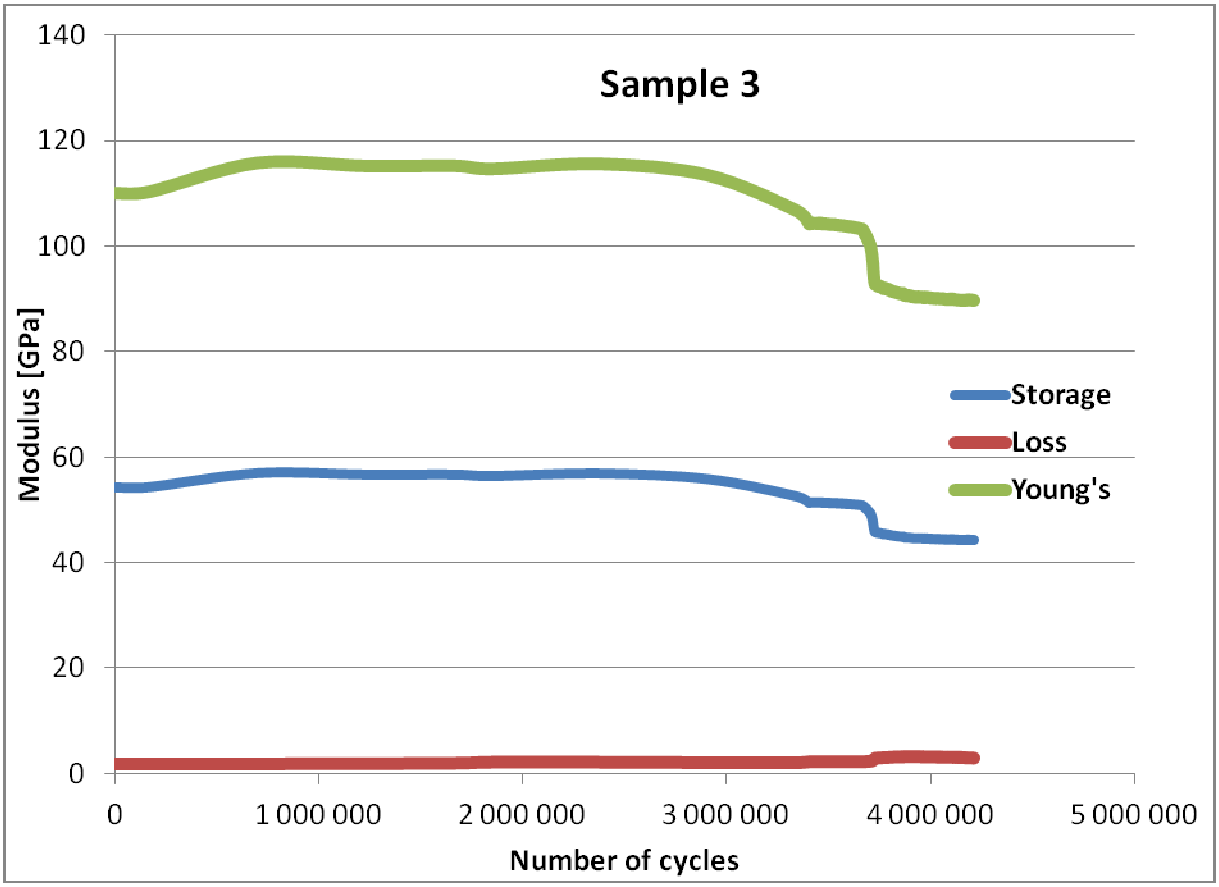

Fig. 4. Results of DMA for sample 3

Figure 1-4 represents modulus-number of cycles relationships registered during DMA analysis. It is possible to observe differences in dynamic properties of investigated samples. Table 4 gathers the numbers of cycles corresponding to the losses of 20% of the Young's modulus and storage modulus. It is possible to note that all CC/ceramic composites represents better dynamic properties in comparison with the CC reference. CC/ceramic composite obtained at 1000 ^{o}C (Sample 1) shows at least 2 times better fatigue life, while CC/ceramic composite obtained at 1500 ^{o}C (Sample 2) shows 5 times better fatigue life respect to the CC reference. CC/ceramic composite obtained at 1700 ^{o}C (Sample 3) exhibits 10 times longer lifetime in comparison to the CC reference.

Composite	Number of cycles when the loss of 20% of the Young's modulus was registered	Number of cycles when the loss of 20% of the storage modulus was registered
Reference	373 000	358 000
Sample 1	1 100 000	767 000
Sample 2	2 020 000	2 042 000
Sample 3	4 050 000	4 120 000

Table 4. Number of cycles when the loss of 20% of the Young's and storage modulus was observed

4 CONCLUSIONS

The aim of this work was to compare dynamic mechanical properties of CC (carbon) composite with CC/ceramic (carbon/ceramic) composites obtained by the impregnation of CC composite with commercially available polysiloxane-based solutions of preceram and their subsequent heat treatment up to 1700 ^{o}C. The results shows that at elevated temperature, CC/ceramic composite obtained at 1700 ^{o}C exhibit the best fatigue properties in an oxidative atmosphere. This results from the presence of silicon carbide that protect carbon fibres and carbon matrix against oxidation.

5 ACKNOWLEDGEMENTS

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